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CURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PA

1a. REPORT SECURITY CLASSIFICATION: UNCLASSIFIED	1b. RESTRICTIVE MA
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION / AVAILABILITY OF REPORT: Approved for public release; distribution unlimited.
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S): SRL-12-F-1995	5. MONITORING ORGANIZATION REPORT NUMBER(S)
6a. NAME OF PERFORMING ORGANIZATION: SCIENCE RESEARCH LABORATORY; 6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION: USAF, AFMC AFOSR/NL
6c. ADDRESS (City, State, and ZIP Code): 15 WARD ST, SOMERVILLE MA 02143	7b. ADDRESS (City, State, and ZIP Code): AFOSR / NL, 110 DUNCAN AVE, STE B115, BOLLING AFB DC 20332 0001
8a. NAME OF FUNDING / SPONSORING ORGANIZATION: BMDO / AFOSR; 8b. OFFICE SYMBOL (If applicable): NL	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: F49620-94-C-0062
8c. ADDRESS (City, State, and ZIP Code): OFFICE OF THE SECRETARY OF DEFENSE, WASHINGTON DC 20301-7100 (110 Duncan Ave, Suite B115 Bolling AFB DC 20332-0001)	10. SOURCE OF FUNDING NUMBERS: PROGRAM ELEMENT NO. 63570E; PROJECT NO. B553; TASK NO. 00; WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)
ADVANCED LOW-COST COMPOSITE CURING WITH HIGH ENERGY ELECTRON BEAMS

12. PERSONAL AUTHOR(S)
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13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
FINAL	FROM 8/1/94 TO 6/30/95	95-07-12	39

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	

Curing of fiber-reinforced polymer composites using high energy electron beams (HEEB) offers impressive advantages for fabricating a variety of military and dual-use applications including composite armored vehicle and automotive structures. HEEB curing greatly reduces the time to cross-link the polymer matrix, while increasing composite tensile strength. The process allows curing at room temperature for high throughput and process flexibility. Electron beam curing also produces stronger, uniform composites with reduced voids and stress concentrations.

Science Research Laboratory (SRL) has developed a new generation of pulsed linear induction accelerators which allow reliable, cost efficient production of high average power electron beams with the necessary parameters for high energy electron beam curing of advanced composites. Unique features of these accelerators include high repetition rate (> 5000 pps), all-solid-state pulsed-power drivers which make these accelerators scaleable to megawatt power levels at a capital cost for the accelerator of less than $3/Watt.

During Phase I, SRL developed novel processes and materials to allow high throughput electron beam composite curing. SRL also developed a costing model for the resin transfer molding/electron beam curing process and applied the model to one high-quantity part. The Phase I results show both technical feasibility and cost effectiveness. During Phase II, SRL will optimize these processes and materials and fabricate composite parts for medium- and high-quantity military applications. SRL will also extend the cost model to lower quantities and additional part categories.

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION	
☐ UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS	UNCLASSIFIED	
22a. NAME OF RESPONSIBLE INDIVIDUAL: Dr Charles Y.C. Lee	22b. TELEPHONE (Include Area Code): (202) 767-5022	22c. OFFICE SYMBOL: NL

DD FORM 1473, 84 MAR — 83 APR edition may be used until exhausted. All other editions are obsolete.

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2 Summary of Phase I Results

The overall objective of the Phase I research program was to develop a novel, cost-effective, automated process for production of composites for a range of military and civilian applications. The process, which combines resin transfer molding with "Tool-free" high energy electron beam curing was demonstrated for the first time during Phase I. Phase I material property and process costing results were very promising, and are summarized in this section.

During Phase II, SRL will extend these results to allow fabrication of full-size parts for three military applications. The cost modeling will also be extended to the low- and medium-production rates characteristic of military parts. The cost modeling will be sufficiently detailed to allow costing of each process step as well as the overall parts cost for a variety of fabrication scenarios.

The Phase I program was divided into four parts: Material Development, Process Development, Material Testing and Technical Cost Modeling. The Phase II program will include these four areas and one additional area: Component Parts Fabrication. The results in each area are presented below in Exective Summary format.

2.1 Executive Summary

- Materials developed for RTM/HEEB composite curing.
  - Novel Resin Systems Synthesized.
  - Resin/fiber sizing (coating) interactions tested.
  - Composite mechanical properties sufficient for target applications.
  - Glass transition temperature (T_g) need additional 20°C improvement
- RTM/HEEB Tool-Free Composite Fabrication Demonstrated For First Time
  - Preforming, RTM, demoulding and curing
  - Composites are handleable solids (B-stage) during curing.
  - Process tradeoffs (temperature, viscosity, gelation) determined.

- Composite and Neat Resin Properties Measured
  - Collaboration with University of Delaware Center for Composite Materials
  - Properties include hardness, T_g, moduli, tensile strength and fiber fragmentation critical aspect ratio.
- Technical Cost Modeling Shows Commercial Potential
  - Automotive floorpan (70 lbs.) used as benchmark
  - Parts are less expensive than conventional steel plus assembly at moderate quantities (<100,000/yr)
  - Cost drivers identified
  - Resin costs should be <$2./lb to be competitive
  - Epoxy (rather than steel) RTM tooling is possible with HEEB Tool-free curing, contributing to lower costs.
- Phase II Program and Experiments Designed
  - Phase II will require additional material and process development.
  - Phase II will also include composite parts fabrication including sections of the composite armored vehicle (CAV) and a HUMVEE hood.

Table II lists three resin systems developed during Phase I which had the necessary properties for Tool-free electron beam curing. Electron-beam-curable resins appropriate for thick section composite applications are not commercially available, and were developed by SRL and its material suppliers for the Phase I research program. The processability and thermal-mechanical properties of these resins were measured as part of the Phase I program.

All of the goals of the Phase I program were met. The physical properties of the fabricated composites met or exceeded design goals. These results are summarized in Table I. The detailed technical cost modeling done in collaboration with the MIT Materials Systems Laboratory demonstrated that the process is cost effective, and determined the process cost drivers.

Measurement	Initial One-Part Resin	Optimized One-Part (STC2-116)	Initial Two-Part (STC2-134)	Target Vinyl Ester Resins 411-C50	8084
Hardness (D-scale)	68	73	84	80	*
Glass Transition Temp. (°C)	59	70	85	105	80
Neat Resin Modulus (10^3psi)	460	510	500	490	460
Resin Elongation to Failure	<2%	12-13%	6%	5-6%	10-12%
Composite Modulus (10^6 psi)	2.4	2.3	2.4	2.5	2.4
Composite Tensile Strength (10^3psi)	40	58	61	56	*
Fiber Fragmentation Critical Aspect Ratio (933 Fiber)	†	57	*	†	58

355 dg 003

Table I: Results of Phase I mechanical and thermal properties measurements. Composite modulus and tensile strength measured at 30% fiber volume fraction. High quality Dow Derakane 411-C50 vinyl-ester resin properties are listed as target values. Note: Initial one-part samples were too brittle to perform this (†) test.

3 Background Information

Glass fiber-reinforced (fiberglass) composites are currently used in a wide range of military applications. Helicopter blades, pressure vessels, aircraft fuel tanks, aircraft flooring, ballistic armor and vehicle panels are just a few of the present applications. The military is expanding the range of applications for composites. Programs are underway to develop composites for large armored vehicles (CAV), for HUMVEE structural components and for lightweight shipping containers.

The benefits of using composites are well known. High strength-to-weight ratios, corrosion resistance and light weight are principal advantages. For armor applications, composites can be effective energy absorbers because of their large fracture surface energy absorption. Figure 1 shows advantages of composite materials which benefit both civilian and military applications. Figure 1(a) shows a weight reduction of one-third curb-weight achieved when steel automotive components were replaced with fiber-reinforced composites in a demonstration Ford vehicle [3]. Figure 1(b) shows the energy dissipation mechanism in composites, whose fragmentation upon impact by a projectile is similar to

Resin	Type	Cure	Viscosity
STC2-116	1-component acrylated urethane	Free-radical	700 cps @ 80°C
STC2-134	2-component polysocyanate + catalyzed acrylated epoxy	Free radical	500 cps @ 40°C
AEB-1	2-component amine + catalyzed epoxy/acrylic blend	Cationic	200 cps @ 20°C

S-2 Glass Sizing	Type	Compatibility
365	Silane-based with film formers and lubricants	Vinyl ester and polyester
449	Silane-based for amine cure	Epoxy
933	Silane-based without film formers and lubricants	High temperature processing; BMI, PEEK

355 dg 002

Table II: Materials used during Phase I. The electron beam curable resins are proprietary formulations developed by SRL and material suppliers. The sizings are available on S2 glass roving from Owens-Corning Company.

a ceramic. The Army has been investigating composite armor for many years, including recent investigation into electron beam cured composites [4].

Conventional composite fabrication techniques incorporate a heat-curing step to cross-link the polymer matrix, converting the resin from a viscous liquid to a stable solid. Heat curing is slow, taking hours or even days for a thick-section composite. Heat curing in an autoclave is the composite fabrication process "bottleneck," limiting throughput. Traditional autoclaving has several other disadvantages: 1) It liberates low molecular weight volatiles in the uncured material, producing bubbles that weaken the composite and cause out-gassing. 2) The system often cures from the outside in, producing uneven shrinkage and causing severe internal stresses. 3) It limits the ability to incorporate heat sensitive materials, such as optical sensors, into the composite.

High energy electron beam (HEEB) curing of polymer matrix composites overcome these limitations. Using advanced high power induction-accelerator technology developed by Science Research Laboratory (SRL), composites can be cured in a few seconds to minutes. HEEB curing occurs at ambient temperature, avoiding thermal gradients, residual stresses and heat-induced distortion. Fast curing ensures that low molecular weight materials polymerize before evaporating, eliminating the production of volatile thermal degradation products.

Component	Weight (lb)		Reduction (lb)
	Steel	CFRP	
Body-in-white	423.0	160.0	263.0
Front End	95.0	30.0	65.0
Frame	283.0	206.0	77.0
Wheels (5)	91.7	49.0	42.7
Hood	49.0	17.2	31.8
Decklid	42.8	14.3	28.5
Doors (4)	141.0	55.5	85.5
Bumpers (2)	123.0	44.0	79.0
Driveshaft	21.1	14.9	6.2
Total Vehicle	3750	2504	1246

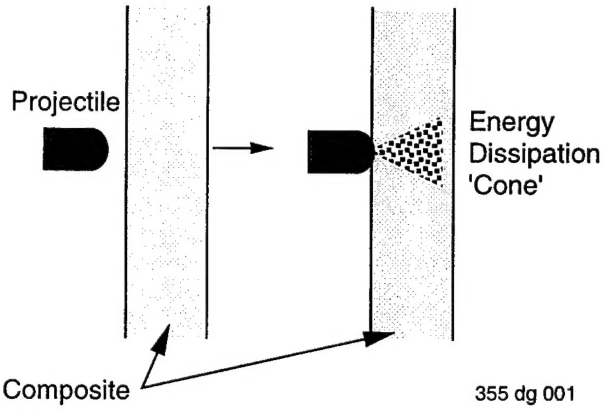


Figure 1: (a) Fiber-reinforced composites reduced curb-weight by one-third in a prototype Ford vehicle. (b) Composite armor spreads impact energy into a high-volume fracture cone.

Reducing the use of volatile organic compounds (VOCs) including solvents which are liberated during curing is an important environmental goal. Curing without solvents is a major reason that radiation-curable inks and coatings are increasingly used. The acrylated urethane and epoxy chemistries of solvent-free coatings and inks are very similar to the materials used in electron beam curable composites. The tool-free composite fabrication process described in this report uses materials which are already handleable solids during curing. The materials crosslink to form strong, hard materials. This is in contrast to conventional heat-curable materials, which react at temperature, releasing gases which include volatile organic materials.

The temperature of composites cured by electron beam rises by 10-50°C during the electron beam curing process. Actually, by adjusting the dose rate, the cure temperature increase can be kept as low as desired. This is in contrast to conventional composite curing, in which the materials are typically heated to 180°C or higher to achieve cure. The lower process temperature allows incorporation of some types of sensor into the material. For example, we have had discussions with Owens-Corning about incorporating their "hollex" fiber (11.5 μm OD, 33% hollow fraction, density 1.9 g/cm^3) into composites with an optical monitoring network hooked up to the fibers to detect microcracking. In fact, a two dimensional weave might allow passively monitored indication (or more advanced – a neural network) showing the approximate location of microcracking. Conventional, high temperature processing tends to break the Hollex fibers. Curing with electron beam at lower temperatures might make this "smart material" possible.

Science Research Laboratory (SRL) has developed a new generation of pulsed linear induction accelerators which allow reliable, cost efficient production of high average power

electron beams with the necessary parameters for curing advanced composites. Unique features of these accelerators include high repetition rate (>5000 pps), all-solid-state pulsed power drivers which make these accelerators scalable to megawatt power levels at a capital cost for the accelerator of approximately $3/Watt.

To be useful for military and civilian vehicle and armor applications, composites must satisfy both cost and mechanical property requirements. As a result of the Phase I effort, SRL has developed novel processes and materials to allow efficient, cost effective HEEB composite curing. A novel composite fabrication system which combines high speed resin transfer molding (RTM) with high energy electron beam curing is described in this report. Raw material costs are minimized by the use of low-cost glass fiber reinforcement and epoxy resins. Processing costs are minimized using the high-throughput RTM/HEEB "Tool-free" process, which can be fully automated.

This Tool-free process was developed by SRL to allow electron beam curing outside the mold. As described in this report, the Tool-free process has significant cost and throughput advantages over other approaches to E-beam curing. Tool-free curing was demonstrated for the first time during the Phase I program.

Strong, durable materials result from the proper choice of matrix resin formulation, fibers, fiber coatings, sizings, and processing conditions during molding and curing. For example, the ability to transfer load from the matrix to the fiber reinforcement depends on good wetting of the fibers and chemical bonding at the fiber/matrix interface. Considerable effort has led to the development of glass fiber sizings optimized for use with traditional heat-curable resins. Testing of several glass-fiber sizings for use with radiation-curable resins was carried out as part of the Phase I program.

In the U.S., the University of Delaware Center for Composite Materials (CCM) has been a leading institution in developing processing methods for conventionally-cured composites and studying the relationship between interface effects and composite properties. Recent work on interface effects in free-radical-cured glass-reinforced epoxy-resin composites is directly applicable to the development of radiation-cure-compatible sizings [5]. CCM personnel also have extensive experience in the measurement of thermal and mechanical properties of composites.

Two material suppliers with considerable experience in formulating radiation-curable resins are Bomar Specialties of Winsted, CT, and Applied Polaramics (API) of Benecia, CA. SRL collaborated with Bomar during Phase I to produce novel resin systems to allow Tool-free E-beam curing. API has worked with Northrop under ARPA's Affordable Polymer Composites Program to produce E-beam curable resins. Both Bomar and API

will be part of the proposed Phase II program, which requires optimization of the novel resin formulations produced during Phase I.

The MIT Material Systems Laboratory (MSL), under the direction of Professor Joel Clark, has developed spread-sheet based costing models for vehicle fabrication. During Phase I, SRL and MSL extended the costing models to include HEEB curing for fabrication of automotive parts. The model showed that the RTM/HEEB Tool-free fabrication process is cost-effective over a broad range of automotive production volumes. The model also identified process cost-drivers. During Phase II the costing model will be extended to military parts and fabrication scenarios, which are centered at production rates below 10,000 units per year.

This program is a collaboration combining SRL's expertise in electron beam curing systems with CCM's experience in composite materials testing, Bomar's and API's experience in resin formulation and MIT MSL's experience in cost modeling. The goal of this collaboration is a new generation of cost-effective, higher-strength composites for both military and civilian applications.

3.1 RTM/HEEB Tool-Free Composite Fabrication

There are several advantages of curing fiber-reinforced polymer-matrix composites with high energy electron beams (HEEB). HEEB curing greatly reduces the time to cross-link the polymer matrix, while increasing composite tensile strength. The process allows curing at room temperature for high throughput and process flexibility. The process releases no volatile gases, and is capable of producing composites with reduced voids and stress concentrations.

The Phase I research was directed at developing materials and processes to fabricate composite components using HEEB curing. Materials include resins, monomers (reactive diluents), reinforcement fibers and fiber sizings (coatings). Although some materials (principally fibers and sizings) are commercially available, other materials (i.e. appropriate resins) are not available, and were developed under the program.

The processes used in this program are resin transfer molding (RTM), vacuum-assisted resin transfer molding (VARTM) and reactive injection molding (RIM), all followed by HEEB curing. The primary applications for the chosen materials and processes are in the areas of structural military and automotive vehicle composites. Glass fiber reinforcement with moderate use-temperature (<300°F) urethane- or epoxy-based matrices are the appropriate materials for these applications. Aerospace applications require more costly, high-strength, high-temperature materials such as carbon-fiber reinforcement and

bismaleimide resins. Work on aerospace-grade materials is also ongoing, as part of the ARPA/Northrop Affordable Polymer Composite Systems program, on which SRL is a subcontractor.

High energy electron beam (HEEB) curing is a line-of-sight process where the uncured resin is exposed to the electron beam either directly or through tooling material such as a vacuum bag or a thin mold window. Each curing option has advantages and disadvantages.

Direct curing without tooling obstacles ("Tool-free" curing) allows the electron beam to cure edges and corners without blockage. It is a more efficient use of the beam since energy is deposited only in the composite, and not into the tool. Direct access also permits curing from both sides of the composite, more than doubling the allowed composite thickness. However, Tool-free curing requires that the uncured composite be a structurely sound, handleable solid, either partially cured to "B" stage or solidified to a crystalline or amorphous state. A RIM process which leaves the composite in a thermoplastic "B" stage for automotive applications was suggested by C. Billiu at Ticom [16]. A direct electron beam curing approach for filament-wound rocket casings was demonstrated at Aerospatiale [1, 17].

Curing through a thin material, such as a 5-mil nylon vacuum bag allows the composite to be tacky during curing. As in direct cure, the issues of handleability and dimension control during curing must be addressed. Military vehicle panels using a hand layup/vacuum bag process have been produced by Damilic Company [18].

Conventional aerospace composites are produced at pressures up to 100 psi and temperatures of 350°F to reduce void content, avoid delamination and maintain dimensional tolerances. For highest strength, low-void parts with the tightest dimensional control, composites can also be HEEB cured while under pressure in a mold. This process requires the electrons to penetrate through significant tooling material.

For HEEB curing, the tooling material thickness (normally 0.25-0.50 inch thick aluminum) must be reduced to allow the electron beam to penetrate to the composite. Electron beam losses in the tool will heat the entire part without contributing to cure. Edges and corners may be difficult to cure without an uneven dose distribution nearby. Thick tooling will be needed on the underside, precluding two-sided curing. For a given beam energy, this will further reduce the allowable part thickness.

In this program, SRL has been investigating direct curing and thin vacuum-bagged curing processes rather than thick-tooled high-pressure processes. This choice is consistent with concentrating on military and automotive vehicle applications rather than high

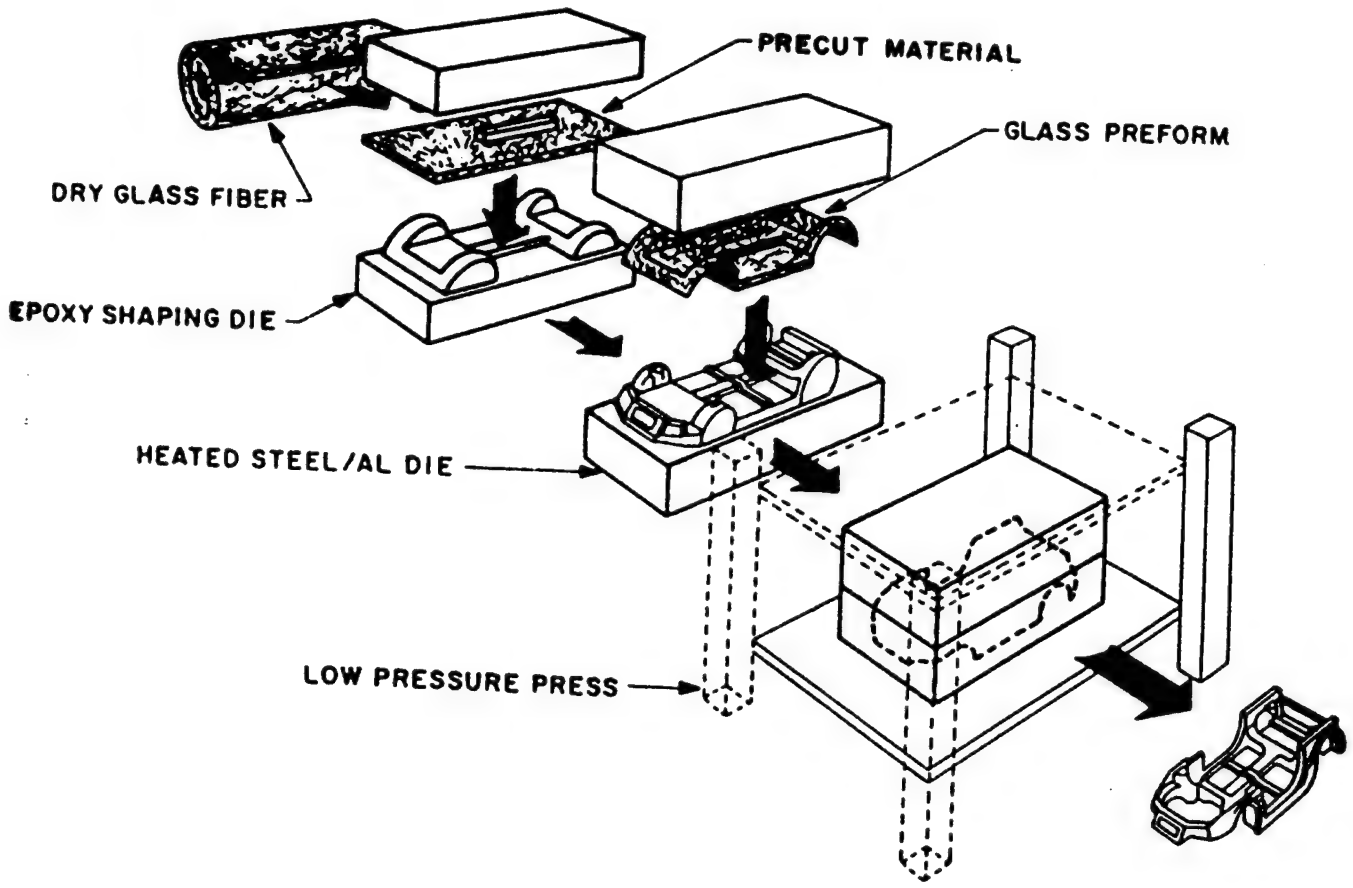


Figure 2: The resin transfer molding (RTM) process.

performance aerospace applications.

Resin transfer molding (RTM) is a composite fabrication process capable of producing complex parts at a high production rate (2 minutes per part per press). The process is shown in Figure 2. In the RTM/HEEB Tool-free process, a preform of oriented glass fibers is mixed with binder and shaped in a preform machine. The preform is transferred to a low pressure, heated press mold. A one- or two-component resin is injected and solidifies in place. After the composite is removed from the mold, it is ready for electron beam curing. Electron beam crosslinking of the composite following RTM forming is used to increase the stiffness (modulus), strength and temperature resistance. Crosslinking results in an inter-connected network of polymer chains caused by additional carbon-carbon bonds.

If a two-component resin containing polyisocyanate + a catalyzed polyol is used in the RTM process, the result is a thermoplastic which can be cured by HEEB into a hard thermoset. The advantage of this technique is the ability to thermally "tack-weld" multiple composite parts together, adding an electron-beam-curable adhesive between the seams [16]. It is even possible to add an acrylated paint, and then cure the frame, adhesive and paint together at one time. The concept is shown in Figure 3. Based on the

available electron beam power from SRL's HEEB equipment, and using the high speed RTM process, such a system will be capable of producing up to 200,000 vehicle frames per year per production line, and allow cost-effective composite fabrication for military and commercial applications.

4 Phase I Experimental Results

The RTM/HEEB Tool-free process was demonstrated for the first time during Phase I. Figure 4 shows composite samples produced during Phase I and Figure 5 shows a schematic of the high energy electron beam curing system used to produce these samples. A picture of the SRL composite curing system used for the Phase I experiments is shown in Figure 6.

4.1 Resin Systems

The most common resin oligomers are ethylene unsaturated polymers including acrylate epoxides, acrylate polyesters and acrylate urethanes. Radiation-curable oligomers and a summary of their properties are listed in Table III.

During Phase I, SRL developed both one- and two- component resin systems for electron beam curing. The one-component systems solidify when cooled to room temperature, and are then available as a handle-able solid for Tool-free curing. The two component systems are liquids which react to also form a handle-able curable solid. In either case, the liquid is directed into a shaping mold containing fiber reinforcement.

The single component resins developed in collaboration with Bomar Specialties Co. are alkoxylated polyols reacted with diisocyanates. A first type are aliphatic-backbone polyols reacted with toluene diisocyanate (TDI) and combined with a monomer such as Tris 2-hydroxyethyl issocyanurate triacrylate (THEICTA) for increased strength and hardness. A second type of resin consist of aromatic-backbone (i.e. Bisphenol A-based) urethanes combined with a monomer such as hexanediol diacrylate (HDDA) to reduce brittleness and viscosity. These resins and monomers (reactive diluents) are liquid at temperatures of 60-100°C and solidify rapidly at room temperature to allow de-molding and curing.

Of the single-component resin systems tested, one of the aliphatic acrylated urethanes, STC2-116, had the highest strength, glass transition temperature and process-ability. Down selection to this resin was made early in the Phase I program. Monomers were added to the resin to increase toughness, since the pure resin system was too brittle.

HEEB Curing of Fiber-Reinforced Composites

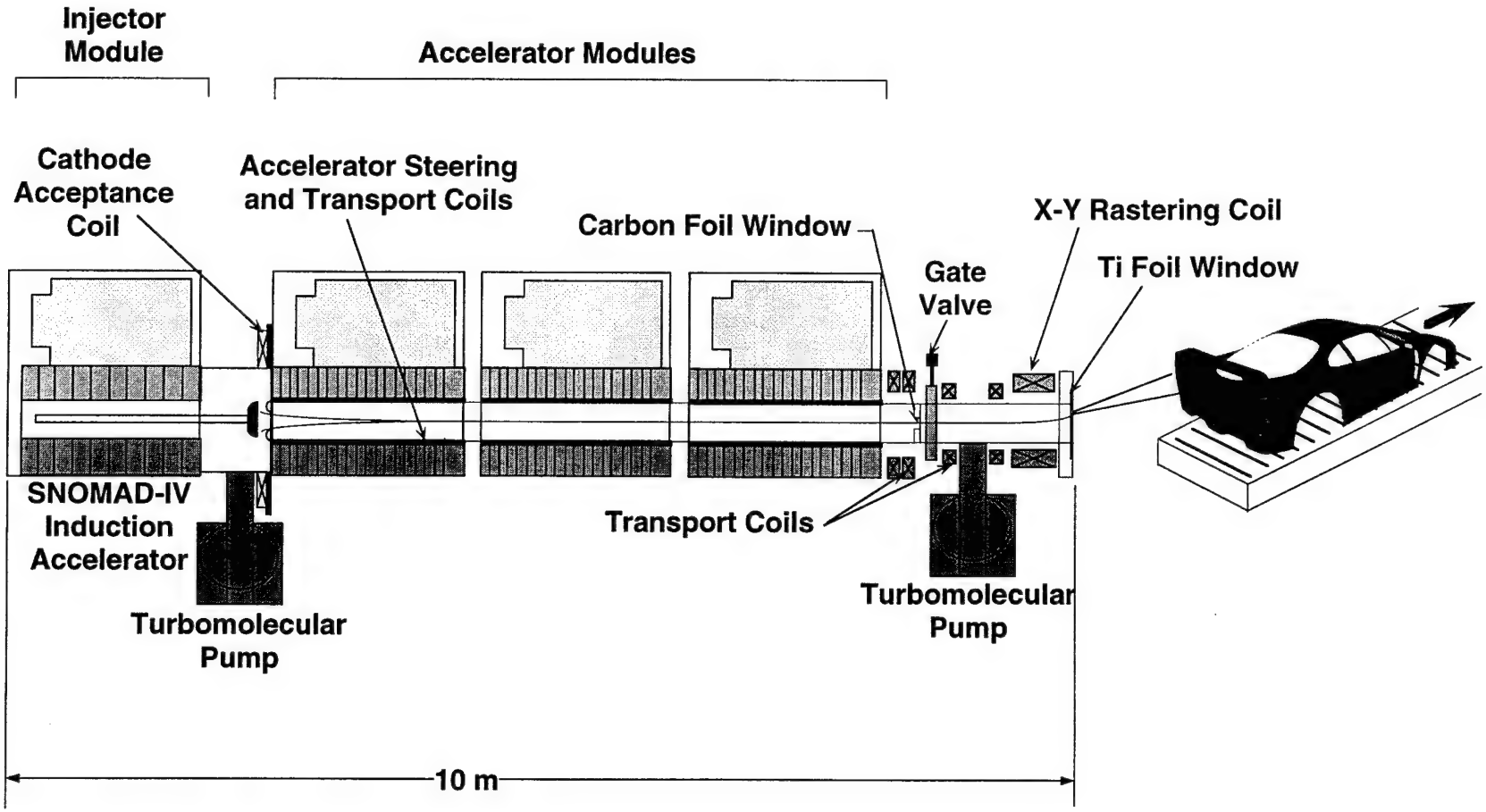


Figure 3: This is the full color schematic with the accelerator and a car.

Four monomers (tetrahydrofurfural acrylate (THFA), phenoxyetyl acrylate (PEA), vinyl pyrrolidone (NVP) and isobornyl acrylate (IBOA)) were tested. The monomer PEA, at a concentration of 5% increased the elongation from under 2% to 12% while maintaining the modulus in the required range above 3 GPa.

Two-component systems were also developed during Phase I. One type of two-phase system is based on the reactions of a polyisocyanate and a catalyzed polyol containing an acrylated epoxy backbone. A variety of catalysts are available to promote the reaction over the reaction time range of 45 seconds to 20 minutes. Of the two-component systems of this type, the resin STC-134b had the best properties.

When irradiated with electron beam, these acrylated resins polymerize by an free-radical addition polymerization reaction. An alternative polymerization mechanism can be initiated by a cationic catalyst such as a diaryl- or triaryl- sulfonium or iodonium salt, which disassociate in the presence of electron beam (EB) or ultraviolet light (UV) [6, 7].

The properties of several free-radical and cationicaly-cured resin systems are shown in Table III. Two advantages of cationically cured systems over free-radical-cured (i.e. acrylated) systems are lower viscosity and shrinkage during cure. However, cationically cured systems are UV sensitive, must be shielded from UV light during storage and handling, and may require a heat post-cure to attain optimal properties.

For this work, the goal was to demonstrate a low-cost ($\leq$ \$2/lb) resin system with the mechanical and thermal properties of a high-quality vinyl ester, and possessing good processability. Good processability includes low viscosity ($\leq$ 500 cps), a long (or infinite) pot life at processing temperature, low toxicity, low shrinkage during cure and insensitivity to oxygen and water vapor. Resin systems developed during Phase I already meet nearly all of these requirements. Some additional resin optimization is planned for Phase II. Optimization will be with respect to thermal properties and durability.

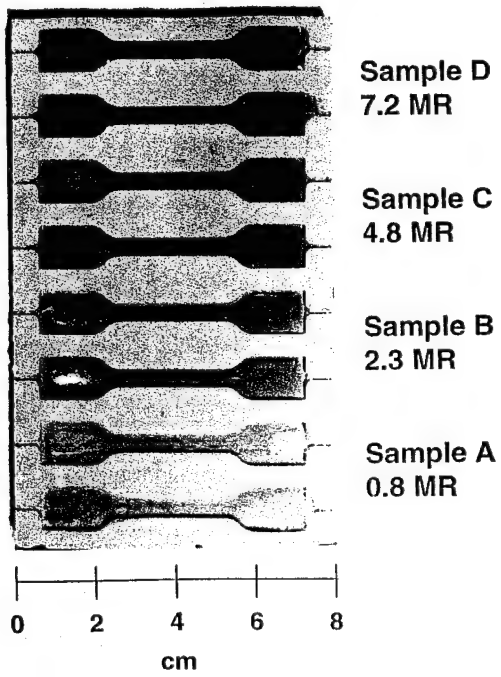
The majority of the Phase I work was conducted using free-radical cure resin systems. The mechanical and thermal results for these resin systems are listed in Table I. At the end of Phase I, work was done using the cationically cured epoxy system AEB-1.

4.2 Fiber/Matrix Interfacial Testing

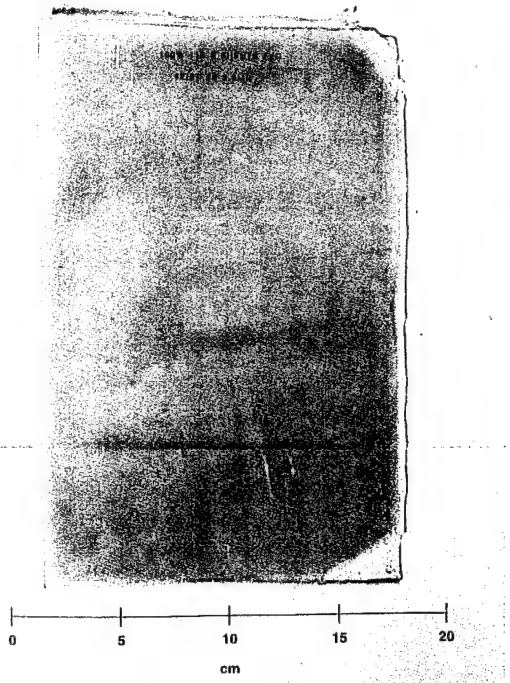
Single fiber fragmentation techniques such as fiber pullout, single fiber fragmentation, and microdebonding have been used extensively for the measurement of fiber/matrix interfacial strength. [11]-[13]. Phase I work utilized the fiber fragmentation method, which produces the largest amount of useful statistical data.

In the fiber fragmentation technique, a single fiber is imbedded axially in a polymer

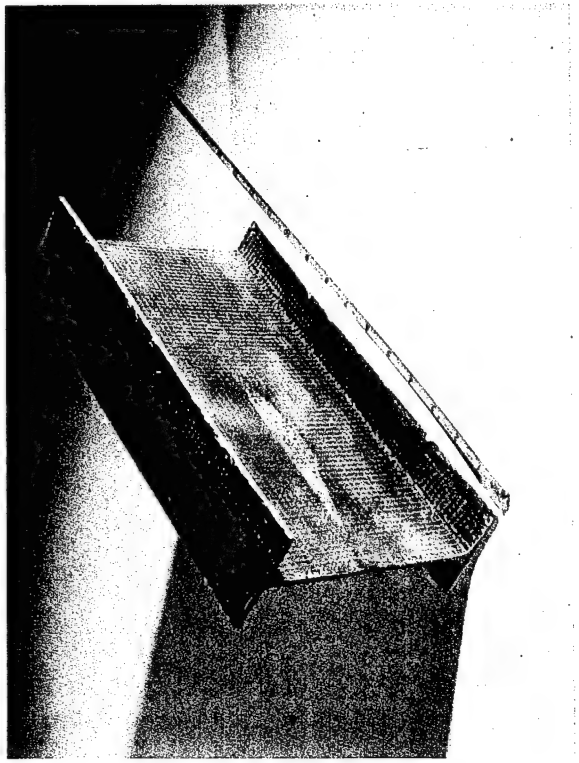
High Energy Electron Beam Cured Composite Samples



"Dogbones" for fiber/ matrix interfacial shear strength test



Tensile test coupon



I-beam for 3-D composite curing demonstration

Figure 4

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The SRL High Energy Electron Beam Composite Curing System

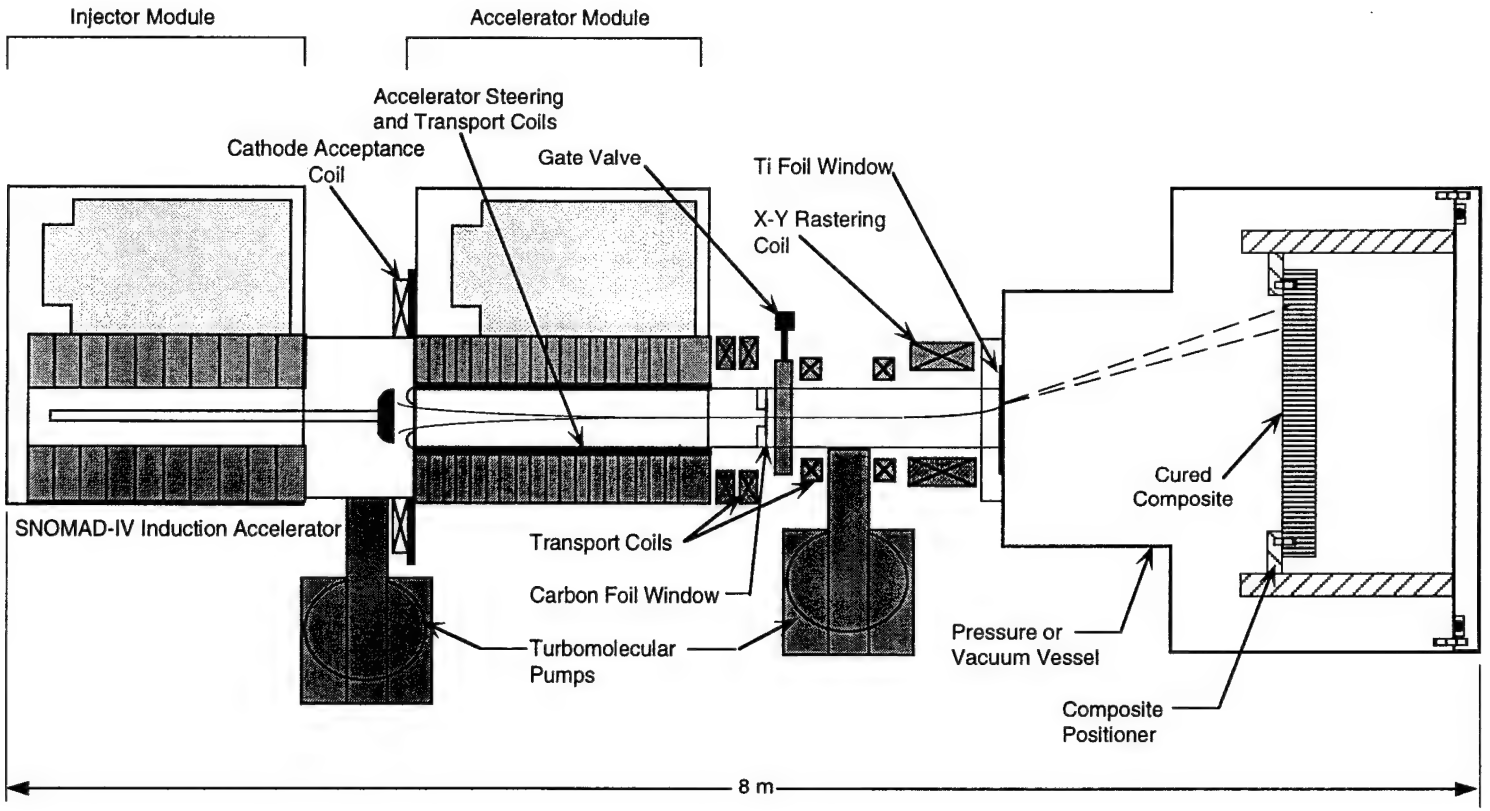

Figure 5: A schematic of the SRL high energy electron beam composite curing system.

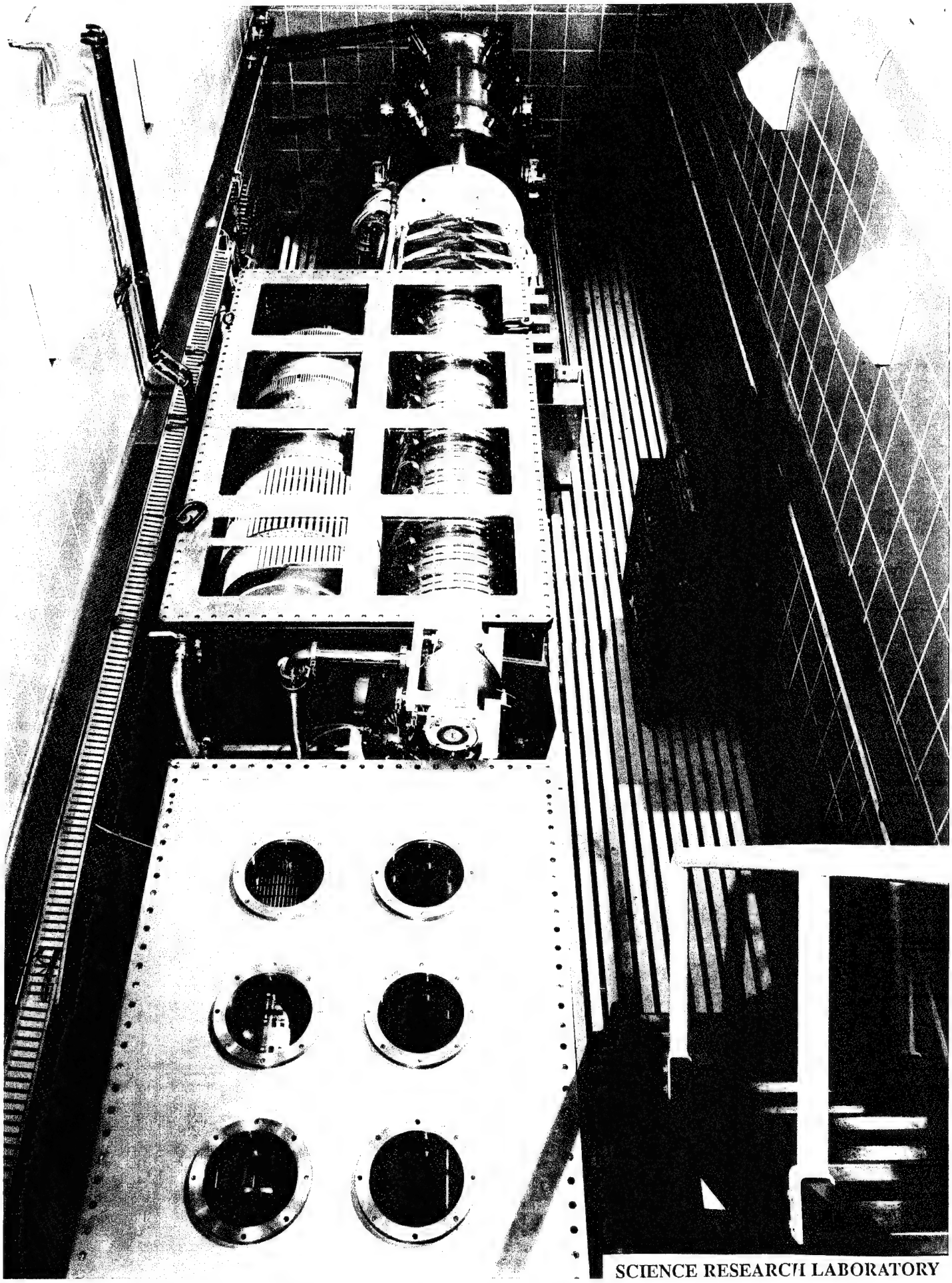

Figure 6: A high energy electron beam composite curing system.

matrix and the specimen is then loaded in tension causing axial shear stress acting on the fiber surface to exceed the fiber tensile strength (σ_f) and cause fragmentation. As the strain increases the fiber continues to fragment until all segments become shorter than a critical length l_c. Beyond this point the interfacial stress acting on the fiber is no longer sufficient to cause fiber breakage and the resulting multifragmented system has reached saturation.

Fiber fragments greater in length than l_c build up stress until the breaking strength of the fiber is attained. At this point, the fiber fractures at the location which possesses the most severe defect hence yielding a distribution of lengths between $l_c/2$ and l_c at saturation. The critical length is calculated by multiplying the mean fragmentation length by a factor of 4/3 [14].

The interfacial shear strength, may be obtained from a force balance using the standard formula: $\tau = 0.5\sigma_f d/l_c$

where σ_f is the fiber strength at critical length l_c and d is the fiber diameter.

Determination of σ_f requires a time consuming statistical treatment. The ratio l_c/d, (known as the critical aspect ratio) is inversely proportional to τ, and is often used to evaluate interfacial behavior. The critical aspect ratio for combinations of resins and fibers was measured during Phase I.

The number and length of each fragment is measured using a microscope equipped with cross-polarizing filters and a calibrated eyepiece. Hence, it is necessary for the matrix material to be transparent and birefringent. For the single fiber fragmentation test to be successful it is also necessary that the matrix material possess a strain to failure two to three times that of the embedded fiber.

For this investigation single fibers were embedded in dog-bone-shaped samples with a 2.5 cm gauge length, 5 mm wide and roughly 2 mm thick. These were prepared by placing single fibers in appropriately designed molds and filling the cavities with resin while maintaining the fibers under tension. Following cure (E-beam or thermal) the samples were demolded, polished and tested. Four to six samples were tested for each fiber-resin combination investigated. A typical fragment size distribution for the tests performed is given in Figure 8.

Owens Corning sized S-2 glass fibers, designated by the manufacturer as 365, 933, 449, were utilized. Fibers were taken from 750 (yards/pound) weight roving provided by Dow Corning. However, the 365 roving was comprised of 13-um diameter filaments, while 933, and 449 were comprised of 9-um diameter filaments. These fiber systems have been sized with coatings claimed to have been designed for use with specific resin systems.

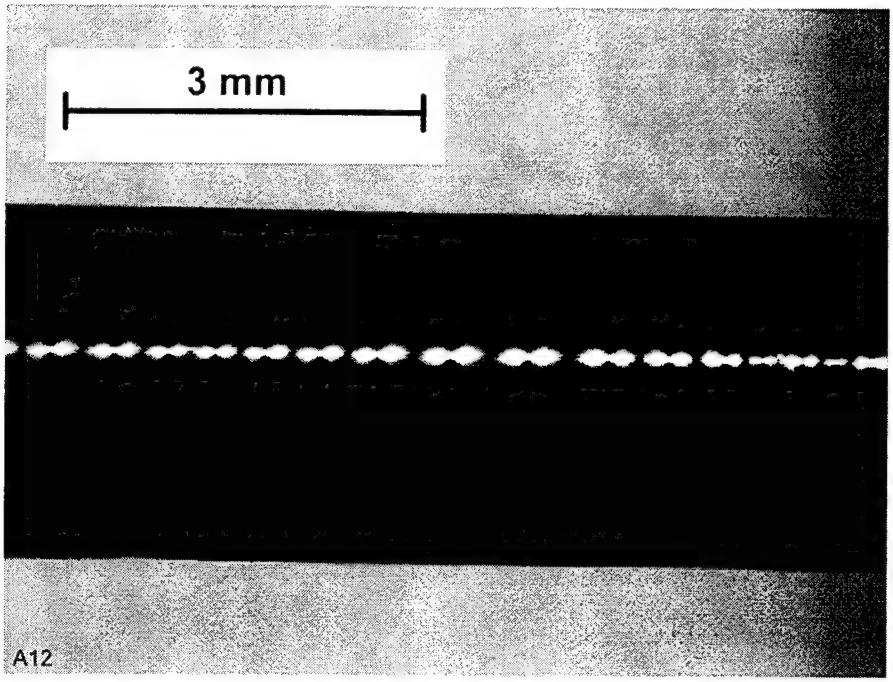

Figure 7: Interfacial shear strength is measured using a fiber fragmentation test. The birefringence pattern of fibers fragments is shown.

The 933 fiber system is designed to be used with high-temperature materials such as BMI and PEEK; 365 with vinyl ester and polyester; and 449 and 463 with epoxy.

Sizing composition was analyzed by stripping the fibers with acetone and measuring Fourier transmission infrared spectroscopy (FTIR) spectra of the dissolved material. Results are shown in Figure 9.

The spectra for the 449, and 365 fibers in Figure 9 show peaks at 1120 cm^{-1}, 1080 cm^{-1}, 940 cm^{-1}, and 3400 cm^{-1}. The peaks at 1120 and 1080 cm^{-1} can be associated with the Si-O-Si asymmetric stretch and Si-O-C asymmetric stretch [8]. The absorption peaks at 940 cm-1 and 3400 cm-1 are due to Si-OH stretch [8]-[10].

The spectrum for 933, however, displays only one of the adsorption peaks associated with silicon-containing species (1120 cm^{-1}). Thus, although all fibers show the presence of silane-derived silicon, the 449, and 356 fibers contain some partially hydrolyzed material, while 933 contains more fully condensed silane. From comparison of the spectra for 449, 365 and 933 with that of Shell Epon 828 epoxy (also in Figure 9) the 463, 449, and 365 contain a substantial amount of un-bound epoxy, which is not seen in the 933 fiber spectrum. These spectra provide a theoretical basis for analyzing the results of interfacial fiber fragmentation tests. These are shown in Figure 10.

Figure 10 shows that there are clear differences between the interfacial behavior of

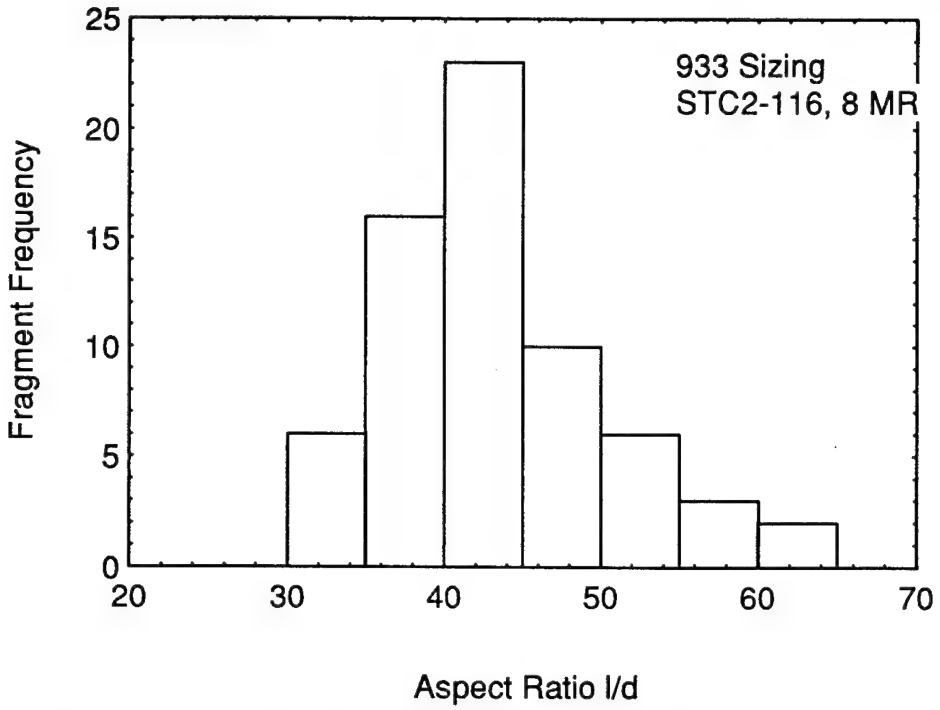

Figure 8: Distribution of fiber sizes in fiber fragmentation testing of interfacial properties. Fragment size length is l and fiber diameter is d.

conventional (heat curable) vinyl ester resin and the electron-beam-curable system STC-116. The vinyl ester resin system achieved maximum compatibility (lowest critical aspect ratio l_c/d) in combination with the 365 fiber system. This was expected because the 365 sizing was designed for vinyl ester composite applications. Maximum compatibility for EB resin STC-116 is with the 933 sizing.

A possible explanation for this difference in interfacial behavior can be found by combining an understanding of the electron beam cure process, the FTIR spectroscopy results and the manufacturer's heat curable processing recommendations. The rapid EB cure may not allow the unbound components of the 449 and 365 systems (seen in Figure 9) time to diffuse into the bulk resin and react with coupling agents. An alternative explanation is that reactivity in the vicinity of the fiber surface may decrease due to particular compounds present in the sizing.

It is clear that differences exist in interfacial behavior of electron beam versus thermally cured systems. These differences may stem from altered cure mechanisms as well as the novel chemistry of these new resin systems. Further investigation into interfacial effects of EB-cure systems is planned for Phase II to elucidate new mechanisms for interface/interphase formation. This work will lead to improved surface treatments for fibers to be used for EB-cured composites.

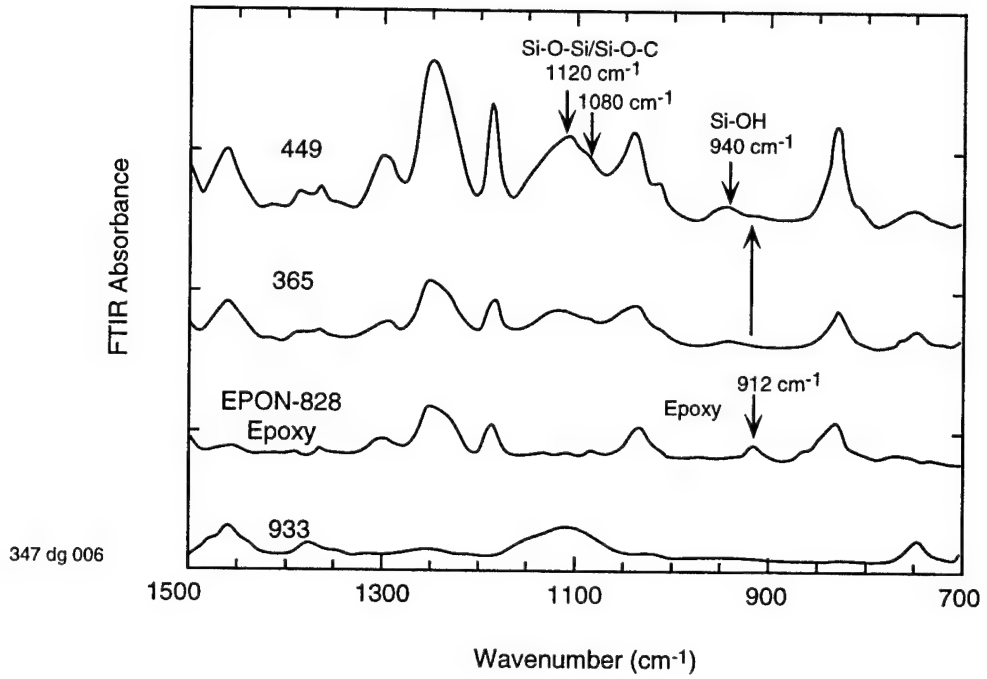

Figure 9: Fourier transmission infrared spectroscopy (FTIR) spectra of fiber sizings. The missing peaks in the 933 spectrum help explain the better interfacial properties for use with electron beam cured resins.

4.3 Mechanical and Thermal Testing

Dynamic mechanical analysis (DMA) was used to measure thermomechanical properties of the resins under development. Temperature scans were performed using a TA instruments 983 DMA on samples having rectangular cross-sections at a rate of 5°C/minute and a fixed frequency of 1 Hz. Figure 11 is a sample DMA trace showing the flexural storage modulus and flexural loss modulus as a function of temperature. For this investigation the glass transition temperature Tg was taken to be the temperature at which the loss modulus attains a maximum. This temperature corresponds to roughly one half the value between the initial and final plateaus found in the flex storage modulus trace.

Values of storage modulus obtained using dynamic mechanical analysis generally agree favorably with flexural modulus values obtained using ASTM techniques provided that the instrument is properly calibrated [15]. Flex moduli reported herein represent values of the flex storage modulus found in DMA traces evaluated at 30°C.

The measured composite and resin moduli are listed in Table I. The measured composite moduli agreed with our theoretical predictions based on measured fiber volume fraction, measured neat resin modulus and compiled glass fiber moduli data.

Phase I material property results are listed in Table I. Hardness was measured with

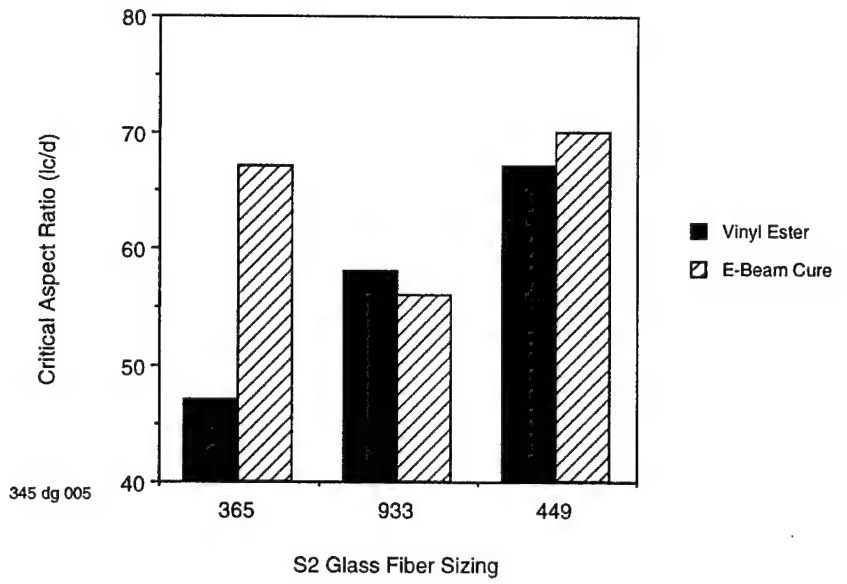

Figure 10: Results of a interfacial shear strength fiber fragmentation test. The interfacial shear strength is inversely proportional to the critical aspect ratio l_c/d. The 933 sizing gives the highest interfacial shear strength and was used to produce composites during Phase I experiments.

a durometer before and after electron beam curing. A composite was considered handleable if it was not tacky and had a durometer reading of 50 (D-scale) before curing. Overhardness before curing was a good predictor of insufficient toughness for one-component resin systems, but less so in two-component systems.

Tensile testing of composites was performed using a modified version of ASTM standard D638 (Specimen Type I Standard Test Method for Tensile Properties of Plastics). Composite test plates manufactured using the Tool-free electron beam curing process were end-tabbed and cut into five 0.75" wide test coupons having nominal six inch gauge length and 0.125" thickness as shown in Figure 12. These were strain-gaged and tested in tension at a rate of 0.05 inches per minute. The reported strength values listed in Table I were obtained by dividing maximum stress by initial sample cross-section and represent averages of the coupons tested. The values of tensile modulus were based the initial linear portion of the stress strain curves.

A summary of tensile test data is listed in Table I. Tensile strengths of the optimized systems exceeded those of the target Dow Derakane 411-C50 vinyl-ester system.

Table I lists values of glass transition temperature and flexural modulus obtained at a dose of approximately 8 MR (80 kGy) for the one component (STC2-116) and two component (134b) resin systems investigated in Phase I. The glass transition temperature

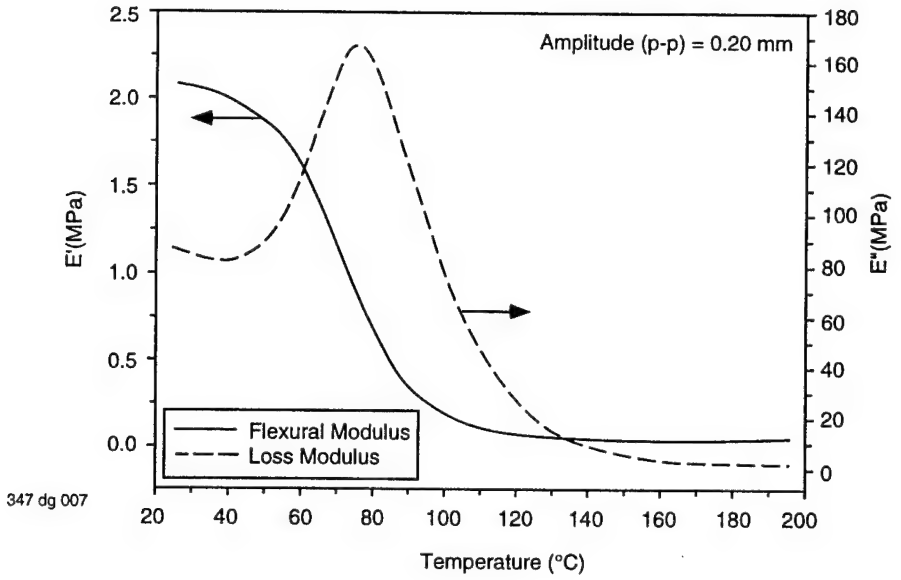

Figure 11: Dynamic Mechanical Analysis (DMA) was used to determine thermo-mechanical properties. The peak of the flexural loss modulus (upper trace) is used to measure the glass transition temperature (T_g).

(a measure of the degree of crosslinking) increases with increasing dose up to a plateau maximum.

For comparison, the PEA modified systems were also cured thermally by the addition of an initiator and heating to 165°C. The thermally cured systems possess higher values of T_g yet possess significantly lower values of strain to failure when compared to the E-beam cured resins. These differences indicate that free radical polymerization initiated by E-beam may yield significant differences in polymer structure compared to thermal initiation.

Acrylated Epoxies	**Hard, solvent resistant**
	Lower cost, $T_g \leq$ 150-200°C
Acrylated Aliphatic Urethanes	**Flexible, tough, non-yellowing**
Acrylated Aromatic Urethanes	**Flexible, low viscosity**
Acrylated Acrylics	**Good weathering properties, Lower T_g**
Acrylated Polyesters	**Low viscosity, flexible**
Cationic-cure Epoxies	**Low viscosity, low shrinkage**

Table III: Radiation-curable resin oligomer selection depends on desired composite properties, fabrication requirements and allowed cost per pound.

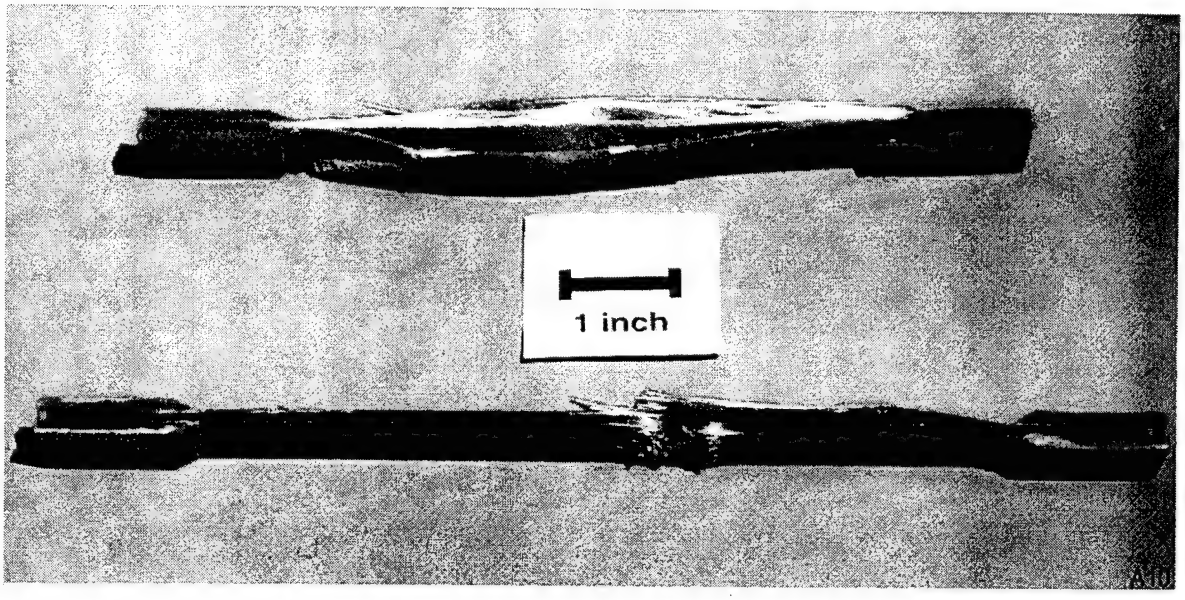

Figure 12: Tensile test coupons cut from a composite fabricated with the Tool-free electron beam curing process. Optimization increased the resin toughness, and changed the fracture mechanism from a brittle delamination failure (top) to a higher-strength fiber rupture near the center of the specimen (bottom).

4.4 Process Cost Modeling

A spread-sheet-based process costing model was developed during Phase I to determine the breakdown of costs for composites fabricated using the RTM/HEEB Tool-free curing process. The model is an extension of work on the costing of automotive fabrication processes by the MIT Material Systems Laboratory (MSL).

This model costs all the materials and processes required for part fabrication. For the RTM/HEEB Tool-Free system, processes include: fiber mat cutting, preform and core molding, preform assembly, resin transfer molding, electron beam curing and inspection. Raw materials include fibers, resins, mats, and catalysts.

The inputs to the model are:

- *Part Specifications* – size, weight, production quantity.
- *Material Choices* – resins, catalysts, reinforcement fiber, fiber mats, cores.
- *Equipment Choices* – accelerator, tooling, presses.
- *Accounting Model* – Dedicated or nondedicated production lines.
- *Exogenous Cost Factors* – wages, electricity, repair and down-time, capital recovery rate.

The database in the model currently data for twenty resins, ten catalysts, six types

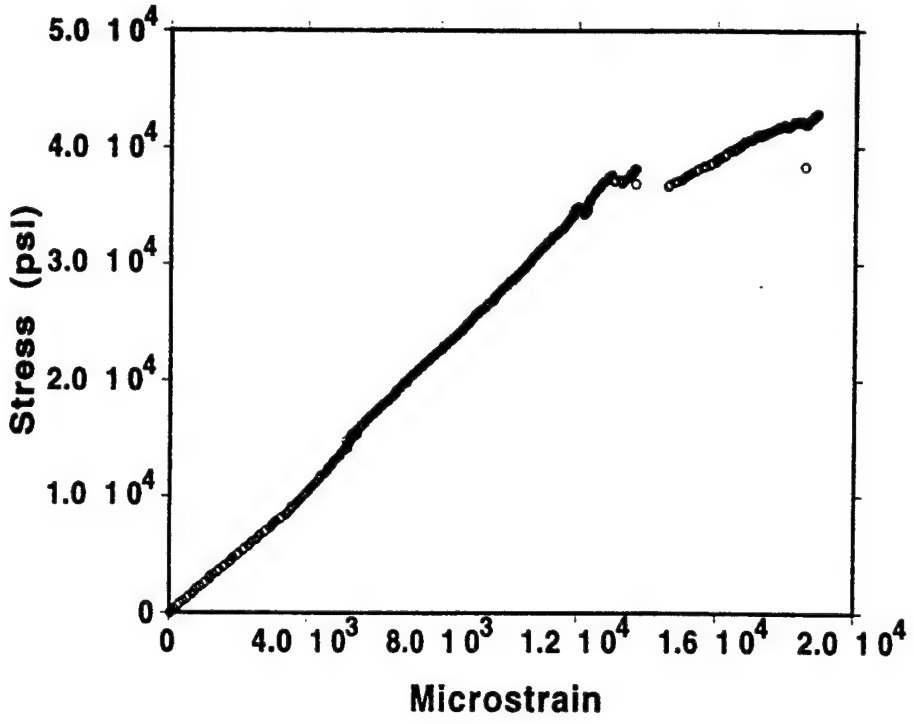

Figure 13: Typical stress-strain curves from tensile testing of electron beam cured composite samples.

of tooling, six accelerators and two part-handling systems.

For each process, the model calculates:

- *Variable Costs* – Equipment cost, tooling cost, building cost, overhead labor cost, installation cost, auxiliary equipment cost, maintenance cost, and capital cost (cost of money).
- *Fixed Costs* – Materials cost, labor cost and energy cost.

Results of the model are shown in Figures 14-16. A 70 lb. automotive floorpan with a large existing costing model database was selected for cost modeling and comparison.

Figure 14 shows the per-unit production cost for this part as a function of yearly production quantity. For quantities of fewer than 150,000 per year, the RTM/HEEB Tool-Free process cost is lower than the steel stamping cost (including assembly). From Figure 14, it can be seen that a large portion of the cost of components fabricated with conventional steel methods is due to the cost of assembly. By making large composites in the RTM/HEEB process, these costs are considerably reduced. Note that the RTM/HEEB per-unit cost remains flat over a wide range of production quantity. This is because tooling and presses for the RTM/HEEB process are inexpensive compared with steel stamping, and because these production quantities fully utilize the electron beam curing facility.

The "blip" in the steel stamping plus assembly cost is due to the need to add a second

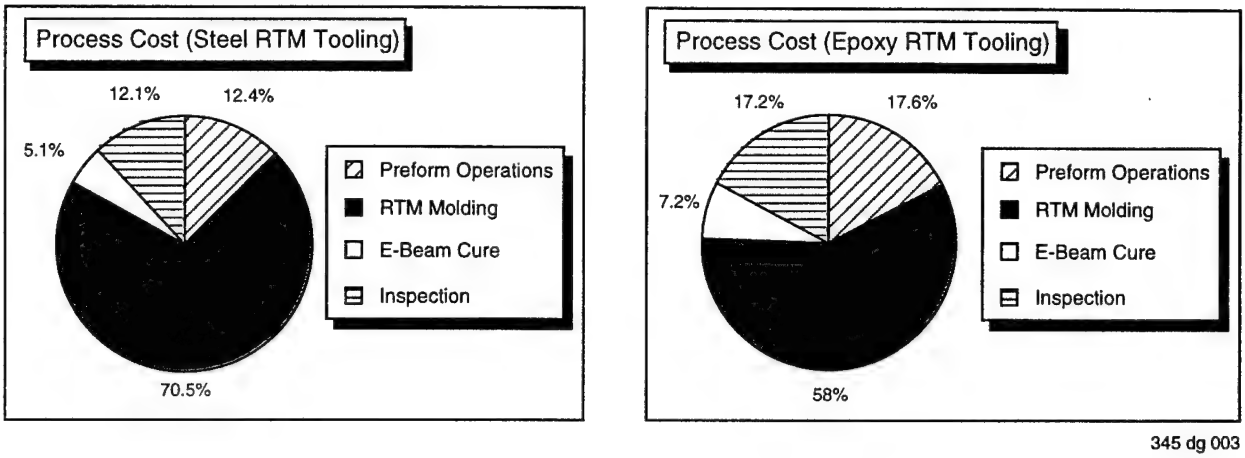

Figure 14: The RTM/HEEB Tool-Free process is cost effective at lower production volumes. This allows for frequent styling changes or lower cost runs of specialty parts such as for military vehicles.

assembly line at production quantities over 200,000 per year. In reality, a manufacturer would probably save money by operating a third shift (overtime) to save the cost of installing a second line.

Figure 15 shows the effect of fiber volume fraction and resin price on part cost. Fiber volume fractions for these components are typically in the 50-65% range. Present low quantity resin prices are between \$2-\$3/lb., giving a part cost of ~\$200. Figure 15 shows that resin price is the most important cost driver for the overall part cost. A Phase II program goal is to reduce resin price below \$2/lb., while attaining material property goals.

Figure 16 shows the relative cost of each RTM/HEEB process. Resin transfer molding constitutes the largest cost fraction (60-70%). The electron beam curing portion is a small fraction of the total process cost (5-7%). Figures 14 and 16 also show that substantial reductions in process cost can be achieved if low cost epoxy tooling can be used in the RTM process.

4.5 SRL Induction Accelerator Technology

Electron beams generated by the SRL linear induction accelerator (LIA) are well suited to high-throughput composite curing. A comparison of the capital cost of the three types of high energy electron beam accelerators and their available power levels are shown in

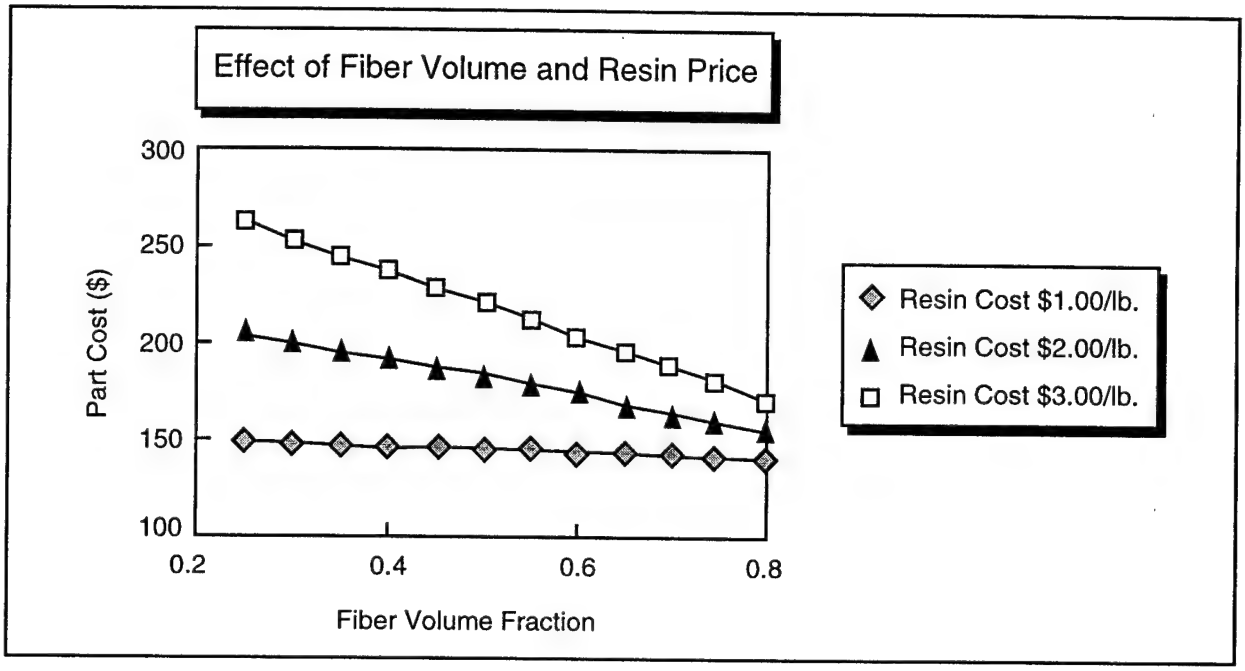

Figure 15: Resin price is the most important cost driver for the overall part cost. Fiber volume fraction is usually 50-65%, and current low-quantity resin prices are between \$2-\$3/lb.

Table IV. High power allows for high-throughput, and cost-effective composite curing. In addition to possessing high average power (MW), beam control is straightforward. The LIA is a pulsed device, capable of low or high repetition rate operation.

Figure 3 shows the components needed for HEEB curing of composites. SRL induction accelerators are modular in design, as shown in Figures 6 and 3. Figure 3 shows four SNOMAD-IV induction accelerator modules, a vacuum system including turbomolecular pumps, magnetic beam transport coils, a hybrid foil/differentially pumped beam extraction system, and a vehicle frame undergoing adhesive bonding and composite curing.

The first accelerator module is an injector containing a thermionic dispenser cathode and grounded anode. The 0.5 MeV electron beam produced in the injector is accelerated to 1.5 MeV in the second accelerator module and by 1.0 MeV in each subsequent module, producing a 3.5 MeV beam at high repetition rate.

The SNOMAD-IV induction accelerator produces a high current ($\simeq$ 500 A) short pulse ($\sim$ 50 ns) electron beam at high repetition rate (5000pps). Accelerator parameters are listed in Table VI and an operating HEEB processing system is shown in Figure 6. The compact, high gradient accelerator modules shown in the figure are driven by all-

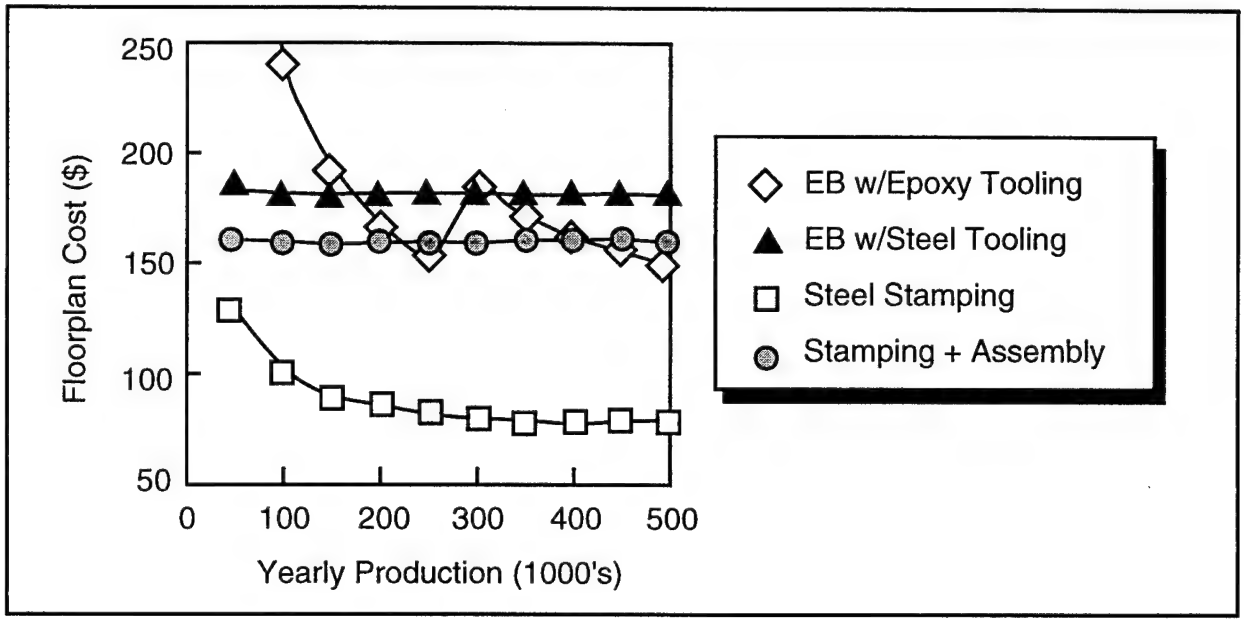

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Figure 16: The relative costs of the RTM/HEEB Tool-Free curing operations. Use of epoxy tooling can reduce the resin transfer molding processing cost. These are process costs, and do not include raw material costs such as fibers and resins.

solid-state, SCR commutated, nonlinear magnetic pulse compressors. The development of these compact, all-solid-state, high repetition rate pulsed drivers was the key innovation leading to reliable, cost-effective linear induction accelerators which are scalable to megawatt power levels and 5 MeV beam energies. A circuit diagram in Figure 17 shows how 500 VDC prime power is converted to 100 kV, 50 ns pulses to drive the electron beam accelerator cells.

As shown in the circuit diagram, 500 VDC power enters through the command resonant charge SCRs which are then switched using the intermediate storage SCRs. The number of SCRs has been selected so that the dI/dt rating of each device can never be exceeded. In addition, the circuit has been designed to protect each SCR under any fault mode condition. The command resonant charging inductors, labeled L_0, isolate the branches of the intermediate storage power supply. The first stage saturable inductor, L_1, is a single turn design which utilizes a 0.6 mil toroidal metglass core. The discharge of the first stage capacitor bank C_1 through L_1 and T_1 to C_2 results in a 5 to 1 temporal compression with a voltage gain of 100.

In the second compression stage, high voltage strontium titanate ceramic capacitors, C_3, are used. Unlike barium titanate, strontium titanate does not suffer from piezoelectric

Operating Parameter	Accelerator Type		
	Induction	Electrostatic	RF
• Maximum Average Current	200 mA	40 mA	5 mA
• Beam Energy	≤ 10 MeV*	≤ 5 MeV	≤ 10 MeV*
• Maximum Beam Power			
1 MeV	200 kW	40 kW	5 kW
3 MeV	600 kW	120 kW	20 kW
5 MeV	1 MW	200 kW	40 kW
• Capital Cost	$3/Watt	$15/Watt	$40/Watt

* Beam Energy ≤ 10 MeV to avoid isotopic activation

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Table IV: A comparison of the three types of high energy electron beam accelerators shows why the SRL linear induction accelerator technology allows for cost-effective composite curing.

mechanical failure under high repetition rate operation. The second stage saturable inductor, L_3 discharges C_3 into a pulse forming network (PFN) with an electrical length of 50 ns. The PFN is a hybrid design consisting of lumped capacitors and distributed inductance. This permits the design to be significantly more compact than either a water-filled transmission line or a lumped element PFN and allows the electrical length to be varied by the addition of capacitive elements. The output saturable inductor, L_4, serves as a passive high speed switch to discharge the PFN into the accelerator cell load. A zinc-nickel ferrite has been selected for the output stage to minimize losses and provide fast switch risetimes. For applications which require a maximally flat $\Delta V < \pm 1\%$ output pulse shape, an additional pulse shaping network, labelled L_p, C_p and R_p, is added before the accelerator cell drive line.

A bias conductor, placed on the axis of the driver, is used for biasing the metglass and ferrite inductor cores into saturation and to provide the reset current between pulses. Both ends of the bias conductor are terminated with a powdered iron core noise suppression inductor. This prevents electrical noise, generated inside the driver, from being transmitted out along the bias conductor.

Each of the SNOMAD-IV driver and accelerator cell assemblies are housed in a rectangular aluminum enclosure which supply cooling and electrical shielding. All high voltage

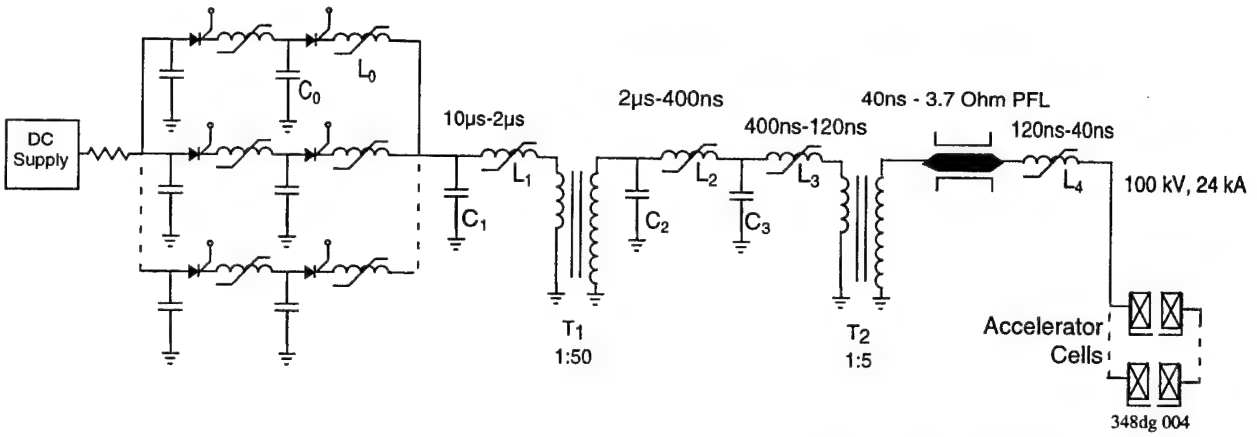

Figure 17: An electrical schematic for the SNOMAD-IV solid state accelerator driver.

bus connections between the driver and induction cells are made within the enclosure. External connections to each module include: 500 volt DC prime power, low voltage timing signals, and entrance and exit ports for the beam.

Several concepts have been investigated for providing cooling and electrical insulation for the driver and accelerator components within the enclosure. Filling the tank with transformer oil permits the accelerator to operate at a repetition rate of 10 kHz for an indefinite period. A water-cooled heat exchanger is used to cool the oil when operating at high repetition rate.

Science Research Laboratory Inc. (SRL) has been actively engaged in the development of induction linear accelerators for high power electron beam production since 1986. Advances in the technology of induction linacs and their associated pulsed power systems by SRL personnel have greatly increased the reliability and shot life of these accelerators, while significantly decreasing the cost per watt of electron beam power delivered by a factor of 5 to 10.

5 Future Work

5.1 Material and Process Development

Many materials to produce composite parts using the RTM/HEEB process are not yet available commercially. For example, several novel resin systems were synthesized and tested during Phase I. The interaction of several fiber sizings (coatings) with these novel resin systems were also tested during Phase I.

The resin systems developed during Phase I were tested and shown to possess the

necessary mechanical properties (modulus, elongation) for the target applications. (The tensile strength of the test composites actually exceeded those of a conventional vinyl ester-based system. Results are summarized in Table I.) However, the glass transition temperature (T_g) of the novel resin systems developed during Phase I are about 20°C lower than the target value. Raising T_g above 100°C is one of the first technical goals of the Phase II program.

Another material development goal is the determination of the optimal fiber sizing for optimum interfacial shear strength. Novel fiber sizings are available for comparison with the currently available commercial sizings.

The RTM/HEEB composite fabrication process was demonstrated for the first time during Phase I and is described in Section 3.1. The process steps include pre-forming operations, resin transfer molding (RTM), demolding and electron beam curing. Pre-forming and RTM processing issues include resin temperature/viscosity and chemical reactivity rates and selection of tooling materials. These processing issues are closely tied to composite material issues. For example, the choice of catalyst (a material issue) affects the reaction rate of the two component resin system. Raising the resin holding tank and mold temperatures to reduce resin viscosity and decrease RTM transfer time also reduces the gel time. Optimization thus involves both materials (resins, catalysts, fiber sizing) and process variables (temperature, pressure, mold geometry).

Many of these process tradeoffs were evident during the Phase I program. For example, if the processing temperature is too low, viscosity limits the resin transfer rate, but process temperatures which are too high may cause reaction to B-stage before complete resin transfer (in two-component resin systems) or gelation in one-component systems. There are also tradeoffs with respect to dose: higher-than-optimum dose curing may give high T_g at the expense of mechanical properties. A Phase II goal is to understand these tradeoffs and to optimize process parameters leading to the selection of the final material systems.

Measurements of material properties during Phase I were primarily conducted at the University of Delaware Center for Composite Materials (CCM). Both neat resin and fabricated composite properties were measured. Properties measured during Phase I included hardness, glass transition temperature, neat resin and composite moduli, tensile strength and fiber fragmentation critical aspect ratio.

Additional measurements to be performed during Phase II include impact and flexural testing. Test methods used during Phase I and additional tests planned for Phase II are listed in Table V. The Phase II goal is to meet or exceed the properties of a high quality

Test	Method
Composite properties:	
Tensile Properties	Tensile Tests
Flexural Properties	3-Point Bend Test
Impact Behavior	Instrumented Impact Tower and Ballistic Tests
Fiber/Matrix Interfacial Shear Strength	Fiber Fragmentation Test
Resin Properties:	
Hardness	Durometer (D Scale)
Glass Transition Temperature (T_g)	Differential Scanning Calorimetry (DSC)
Modulus	Dynamic Mechanical Analysis (DMA)

Table V: Material properties tests for Phase II.

conventional (DOW Derakane) vinyl ester resin composite in all material categories. These properties are listed in Table I.

Technical cost modeling during Phase I concentrated on high throughput automotive applications. A 70 lb. automotive floorpan was used as a benchmark case. Phase II cost modeling will concentrate primarily on military applications. The goal for Phase II is to extend the cost modeling to lower volumes and to focus on military parts and components.

An important goal of the Phase II program is to fabricate prototype parts which can be compared with composite parts produced with conventional processes. Three types of components for three military applications will be fabricated. Components to be fabricated include representative sections of the Composite Armored Vehicle (CAV) lower hull, a HUMVEE hood and a composite shipping container. These are described in more detail in the next section.

5.2 Planned Phase II Experiments

The Phase II experiments and program are designed to optimize the processes and materials developed during Phase I and use the resin transfer molding / electron beam (RTM/HEEB) curing Tool-free process to fabricate components for military and dual-use military/civilian applications. The RTM/HEEB Tool-free process is described in Section 3.1. Section 4 contains the results of the Phase I program, with emphasis on the additional work required to attain the Phase II goals. The novel high energy electron beam curing technology developed by SRL is described in Section 4.5.

Future work during Phase II will be divided into the following areas: Materials Optimization, Process Optimization, Materials Properties Testing, Technical Cost Modeling

Parameter	Phase II Experiments	System Design
Energy (MeV)	1.5, 5, 10	$\leq$10
Average Power (kW)	1-50	$\leq$500
Dose (MR)	4-15	$\sim$12
Cure Temperature Rise (°C)	20-80	<50
Part Thickness (cm)	0.5-2	≤ 4
Process Rate (lb/min)	NA	$\leq$100
Resin Injection Temperature (°C)	20-70	$\leq$50
Tooling Temperature (°C)	20-50	$\leq$50
Tooling Material	Epoxy, Teflon, Al	Epoxy
Injection Pressure (psi)	10-50	$\leq$40
Processing Atmosphere	Air, Inert Gas	Air or CO_2 Blanket

Table VI: Phase II experimental and full-scale system design parameters.

and Composite Parts Fabrication.

Materials Optimization –

SRL will optimize resins and fiber sizings developed during Phase I with respect to both material properties and cost. The goal for this task will be physical properties exceeding those of high quality vinyl ester composites, at a resin price of less than \$2/lb. Both one- and two-component resin systems will be optimized as required for the various applications. Fiber sizings will be modified as needed to optimize performance, especially flexural strength.

Process Optimization –

The RTM/HEEB Tool-free curing process will be optimized to attain the target mechanical properties in test specimens and also for full-size prototype components. For each material system (resin, fiber, sizing), optimized process variables including resin and mold temperature and fill pressure, tooling geometry, tooling material, electron beam curing dose, dose rate, curing atmosphere and curing pressure will be determined.

Material Property Testing –

Test samples at the volume fractions typical of vehicle panels (50%-65%) will be fabricated using the RTM/HEEB Tool-Free process. Sample testing will include tensile strength, modulus, glass transition temperature, flexural strength, fiber fragmentation

/ interfacial shear strength and impact behavior. Table V lists materials property tests and methods planned for Phase II.

Technical Cost Modeling –

SRL will conduct cost-benefit analyses of the RTM/HEEB Tool-free process for manufacturing components at low-, medium- and high-rates for military and dual-use applications. The analysis will extend the spread-sheet-based model developed during Phase I to lower vehicle production rate, comparing Tool-free curing with In-Tool HEEB curing and with steel stamping. The modeling will allow for a mixture of part types (i.e. HUMVEE panels, hoods, armor, etc.) together with single or multiple dedicated or non-dedicated curing lines. A combination military vehicle / commercial vehicle production line will also be costed. Technical cost modeling will be done in collaboration with personnel from the MIT Material Systems Laboratory, under the direction of Professor Joel Clark.

Composite Parts Fabrication –

SRL will fabricate three types of composite parts for military and dual-use applications. These parts include a composite shipping container and representative sections of a HUMVEE hood and a composite armored vehicle (CAV) structural part. Preliminary discussions with CAV personnel have identified a candidate part which is amenable to fabrication with the RTM/HEEB process. The part is the CAV lower hull and is shown in Figures 18(a) and (b). The lower hull is a 67 inch-long, boat-shaped section with 0.2inch skin and a 1.5 inch foam core. As the part curves to support the sidewall near the roadwheels (See Figure 18(b)) it expands to a maximum 0.5 inch thickness, which is consistent with HEEB curing.

Fabrication of advanced radiation-curable composites and composite curing experiments will be performed using the existing high energy electron beam (HEEB) material processing facility at Science Research Laboratory, shown in Figure 6. The composite curing parameters for this system are listed in Table VI. Also listed are full-scale system design parameters.

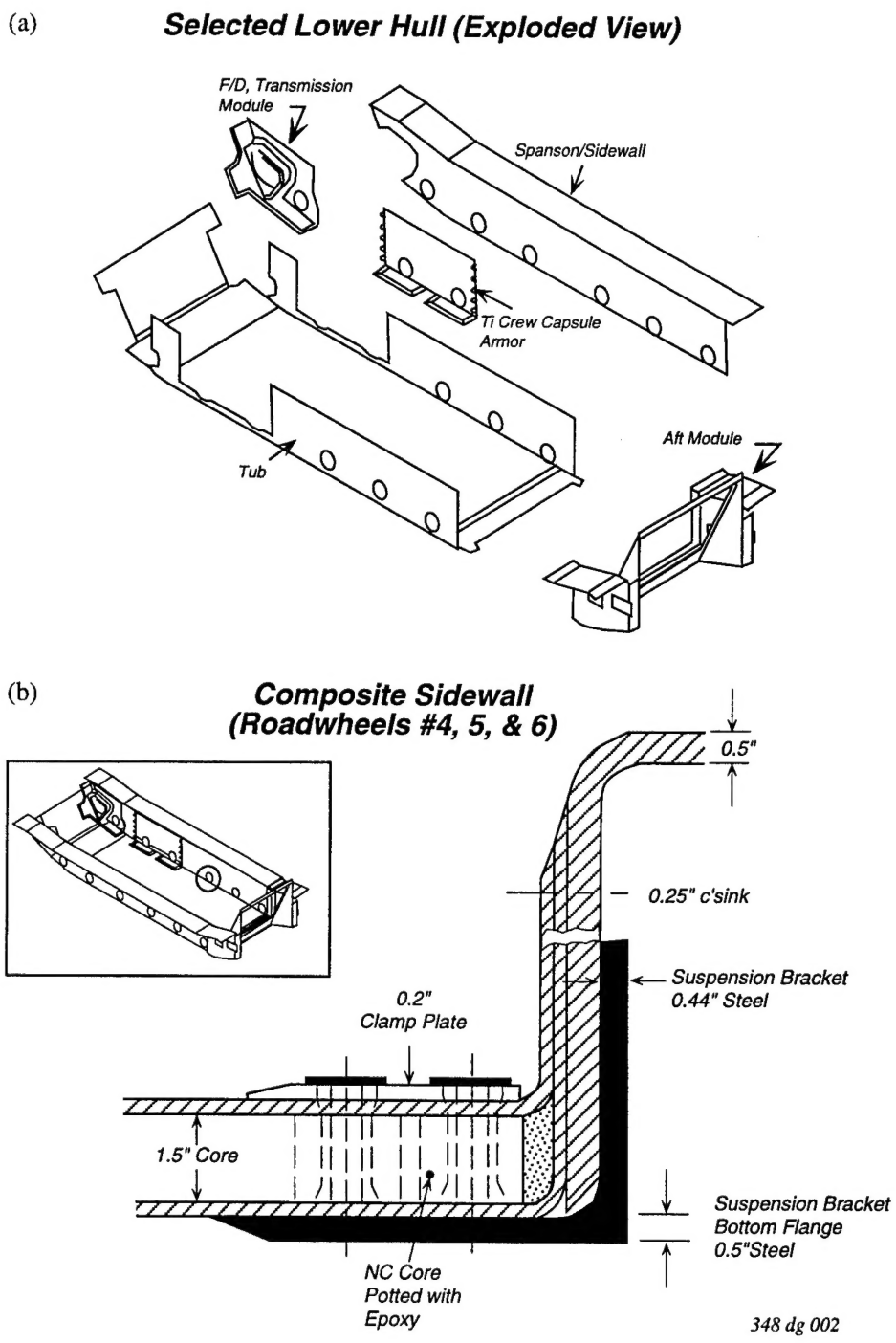

Figure 18: Representative sections of the Composite Armored Vehicle (CAV) Lower Hull will be fabricated using the RTM/HEEB Tool-free process during Phase II. (a) Assembly drawing. (b) Details near the roadwheels.

References

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